



UNIVERSITI PUTRA MALAYSIA

**EFFECTS OF ELECTRON BEAM IRRADIATION ON THE
MECHANICAL PROPERTIES OF ETHYLENE OCTENE COPOLYMER,
POLYPROPYLENE AND TRIMETHYLOLPROPANE TRIACRYLATE
BLENDS**

HARRIS C. RAJ KUMAR.

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By

HARRIS C. RAJ KUMAR

**Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of Master of Science**

July 2005



DEDICATION

This thesis is dedicated to my beloved wife, Jayashree Kanniah.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

EFFECTS OF ELECTRON BEAM IRRADIATION ON THE MECHANICAL PROPERTIES OF ETHYLENE-OCTENE COPOLYMER, POLYPROPYLENE AND TRIMETHYLOLPROPANE TRIACRYLATE BLENDS

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July 2005

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The effect of electron beam irradiation on blends of ethylene-octene copolymer (EOC) and polypropylene (PP) with and without trimethylolpropane triacrylate (TMPTA) at various ratios was studied. Initial study showed that specimens with 90 wt% PP : 10 wt% EOC displayed optimum tensile strength of 29.12 MPa at 40 kGy. Samples of this ratio were then blended with 1, 2 and 3 wt% TMPTA. There was a significant increase in gel content as the TMPTA increased crosslink density in the samples, compared to samples without TMPTA. The plasticizing of TMPTA was obvious during the tensile tests. Furthermore, TMPTA caused the system to break into smaller networks as a result of increasing number of radicals. However, the sample with 3 wt% TMPTA exhibited the highest tensile strength (30.46 MPa) at 40 kGy dose of irradiation. This was also proven in the tensile modulus test. As expected the elongation at break test indicated a decline in values as higher crosslink density decreased the chain mobility, thus reducing elongation. Further tests on samples with 90 wt% PP : 10 wt% EOC : 3 wt% TMPTA were also carried out. The hardness of the samples was quite stable throughout all irradiation doses. However, the impact test displayed a gradual decrease from 73.13 J/m at 0 kGy with increasing irradiation dose. Finally, the flexural strength and flexural modulus displayed optimum properties at 10 kGy irradiation of 39.89 MPa.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**KESAN SINARAN ELEKTRON TERHADAP SIFAT MEKANIKAL ADUNAN
KOPOLIMER ETILENA-OKTENA, POLIPROPILENA DAN
TRIMETILOLPROPANA TRIAKRILAT**

Oleh

HARRIS C. RAJ KUMAR

July 2005

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Kesan sinaran elektron terhadap adunan kopolimer etilena-oktena (EOC) dan polipropilena (PP) dengan dan tanpa trimetilolpropana triakrilat (TMPTA) pada nisbah berlainan dikaji. Kajian menunjukkan bahawa sampel dengan 90 %jisim PP : 10 %jisim EOC mempunyai nilai tensil tertinggi iaitu 29.12 MPa pada 40 kGy. Sampel dengan nisbah ini kemudiannya diadun dengan 1, 2 dan 3 %jisim TMPTA. Peningkatan yang jelas dikesan dalam kandungan gel kerana TMPTA menaikkan ketumpatan tautsilang, berbanding dengan sampel tanpa TMPTA. Kesan pemplastikan TMPTA jelas semasa ujian tensil. Tambahan pula, TMPTA menyebabkan sistem pecah kepada rangkaian yang lebih kecil akibat peningkatan dalam bilangan radikal bebas. Walau bagaimanapun, sampel dengan 3 %jisim TMPTA menunjukkan nilai tegangan yang tertinggi (30.46 MPa) pada 40 kGy sinaran. Ini juga terbukti semasa ujian modulus tegangan. Seperti yang dijangka, ujian pemanjangan menunjukkan pengurangan dalam nilai akibat peningkatan dalam ketumpatan tautsilang yang mengurangkan pergerakan rantai. Ujian selanjutnya telah dilakukan pada sampel dengan 90 %jisim PP : 10 %jisim EOC : 3 %jisim TMPTA. Kekerasan sampel agak stabil pada semua dos sinaran. Walau bagaimanapun, ujian hentaman menunjukkan pengurangan dari 73.13 J/m pada 0 kGy dengan peningkatan dos sinaran. Akhir sekali, kekuatan kelenturan dan modulus kelenturan menunjukkan nilai optimum pada dos 10 kGy sinaran dengan kekuatan 39.89 MPa.

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I certify that an Examination Committee met on 20th July 2005 to conduct the final examination of Harris C. Raj Kumar on his Master of Science thesis entitled “Effects of Electron Beam Irradiation on the Mechanical Properties of Ethylene-Octene Copolymer, Polypropylene and Trimethylolpropane Triacrylate Blends” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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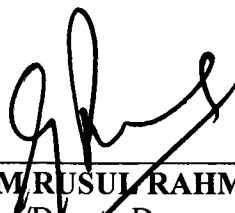
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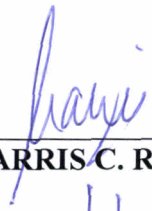
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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



HARRIS C. RAJ KUMAR
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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BS	British Standard
EOC	Ethylene-octene copolymer
HDPE	High density polyethylene
IRHD	International Rubber Hardness Degrees
ISO	International Standard Organisation
LDPE	Low density polyethylene
LLDPE	Linear low density polyethylene
PE	Polyethylene
PEE	Polyethylene elastomer
PP	Polypropylene
RIC	Radiation-induced conductivity
TMPTA	Trimethylolpropane triacrylate
VLDPE	Very low density polyethylene

CHAPTER 1

INTRODUCTION

1.1 Polymers

Polymers are actually large molecules, made up of simple repeating units. Derived from the Greek language, '*poly*' means many and '*mer*' means part. These repeating units can be of the same or different types. These repeating units are called monomers.

Polymers, in the industrial world, can be categorized into three classes. These are plastics, fibers and rubbers (elastomers). The classification of polymers into these classes is based largely on a particular mechanical property called modulus (in other words stiffness). Fibers have the highest modulus and rubbers the lowest (Stevens, 1990).

Rubbers, or elastomers, are polymers that exhibit resilience, or the ability to stretch and retract rapidly. Mostly have a network structure. Natural rubber exists in different forms, but by far it is composed entirely of *cis*-1,4-polyisoprene. Synthetic rubber, the production of which surpassed that of natural rubber in the 1950's, comes in a variety of forms. Styrene-butadiene, polybutadiene, and ethylene-propylene account for about 70% of total production (Morton, 1973).



1.2 Polyethylene

Polyethylene is an olefin polymer manufactured in the largest tonnage of all the thermoplastic materials. The sources of ethylene and other olefins are petroleum and natural gas (Haley and Turner, 1975). They are produced by the addition polymerization of ethylene to give the basic structure – $(\text{CH}_2 - \text{CH}_2)_n$ -.

The ultimate properties of polyethylenes are influenced by a number of structural and morphological factors such as type of comonomer(s), amount of comonomer(s), distribution of comonomer(s), degree of branching, degree of crystallinity, average molecular weight and molecular weight distribution (Simanke *et al.*, 1999). For simplicity, the overall effect of these influences is broadly quantified by the density of polyethylene, which generally ranges from 0.85 to 0.98 g/cm³.

1.2.1 Ethylene-octene copolymers (EOC)

Copolymers of ethylene and another 1-olefin made using metallocene catalysts show significant differences from conventional LDPE and LLDPE that can be used advantageously in applications. First, these copolymers possess narrow molecular weight distributions compared to traditional polyethylenes. Second, the metallocene catalyst produces polymers with uniform comonomer content incorporated along the backbone. This uniformity in polymer structure, consisting of a few long-chain branches and a controlled amount of short-chain branching along the backbone eliminates the problems of mechanical integrity and processability that are associated with LDPE and LLDPE, respectively (Hurley *et al.*, 1996). Unlike traditional

polyethylenes, these copolymers possess negligible crystallinity (Walton and Karande, 1996).

Studies have shown that ethylene-octene copolymers have good tear resistance and long shelf life, making them good packaging materials (Bensason *et al.*, 1997). In particular, ethylene-octene copolymer has been shown to provide a higher toughening contribution than either the ethylene-propylene or ethylene-butene copolymer.

1.3 Polypropylene

Polypropylene (PP) is a semi-crystalline polymer with a molecular weight in the range of 200 000 – 600 000. The chains in the amorphous region of polypropylene are in random coil conformation and the chains in the crystalline region are in a helical conformation.

Polypropylene possesses exceptional properties including excellent chemical and moisture resistance, good ductility and stiffness, and low density. It is also easily processed and inexpensive. The properties of polypropylene are limited by its low impact resistance (Phan and Denicola, 1998), particularly at low temperatures, due to its high glass transition temperature and relatively high degree of crystallinity.

1.4 Crosslinking Agents

Crosslinking agents are monomers with difunctional or multifunctional groups. It is used to encourage crosslinking among polymer chain, thus enhancing and increasing the three-dimensional network density. Understanding how difunctional and multifunctional monomers influence the properties of various oligomers is crucial to successful formulating. Monomers greatly affect both performance and physical properties - including viscosity, surface cure, chemical resistance, pendulum hardness, flexibility, and stain resistance. With careful monomer selection, formulators can manipulate the final properties of their coatings, inks and adhesives for optimal performance.

1.5 Polymer Blends

Polymers are blended to achieve a micro heterogeneous mixture of two or more polymers to improve the mechanical, environmental and rheological properties. This area offers the development of new and improved materials with enhanced and specified combination of properties. Some blends are also prepared due to economical reasons. Even so, some polymers are not miscible and require the introduction of compatibilizers.

Polymer blends are prepared by mechanically mixing the melts, lattices or different polymer solutions. Another method would be the polymerization of a monomer in the presence of another polymer.

1.6 Scope of Research

As ethylene-octene copolymers are relatively new and their properties have not been identified yet, research on this material is necessary to determine the uses for the local market. In countries such as the United States of America, the α -olefin copolymers has been extensively used for various purposes such as piping, tubing, medical apparatus, rubber lining, weather strips and so on. A study on the mechanical properties when blended with a plastic, namely polypropylene and a crosslinking agent, TMPTA, with and without irradiation, will provide an idea of the scope of application where this material can be used. By blending the two polymers, the rigid properties of polypropylene and the low tensile property of the ethylene-octene copolymer can be improved by blending the polymers. In previous research, irradiating the ethylene-octene copolymer has significantly improved the tensile strength. As such, it would be plausible to study the effect of irradiation on the polypropylene and ethylene-octene copolymer blends.

1.6.1 Objectives

The objectives of this research are to:

1. Characterize the mechanical properties of ethylene-octene copolymer and polypropylene blends with and without trimethylolpropane triacrylate.
2. Optimize blend parameters for ethylene-octene copolymer, polypropylene and trimethylolpropane triacrylate blends.
3. Study the effects of electron beam irradiation on ethylene-octene copolymer, polypropylene and trimethylolpropane triacrylate blends.

CHAPTER 2

LITERATURE REVIEW

2.1 Polyethylene

2.1.1 Types of Polyethylene

High density polyethylene (HDPE) are basically homopolymers in the range of 0.960 - 0.970 g/cm³ and are prepared using coordination catalyst, e.g. Ziegler-Natta type catalysts, at low or moderate pressures.

Low density polyethylene (LDPE) are branched homopolymers having densities in the range of 0.915 – 0.930 g/cm³ as well as polar groups resulting from copolymerization, e.g. with vinyl acetate or ethyl acetate. LDPE is prepared at high pressures using free radical initiators. LDPEs contain relatively large number of long chain branches extending from the backbone with alkyl substituents of 2-8 carbon atoms (Kontou *et al.*, 2002).

Linear low density polyethylene (LLDPE) is prepared at low pressure but incorporates a relatively higher amount of an α -olefin comonomer such as 1-butene, 1-hexene or 1-octene, using Ziegler-Natta or metallocene catalysts, to introduce enough short chain branches into the linear polymer to reduce the density of the resultant polymer to about 0.915 – 0.930 g/cm³. LLDPEs have thinner lamellae and smaller spherulites (Bensason *et al.*, 1996). LLDPEs are characterized by little long chain branching. The LLDPE structure has a linear backbone, but it has short,

uniform branches that prevent the polymer chains from packing closely to each other. The advantages of LLDPE are that the polymerization conditions are less energy-intensive and the polymer's properties may be altered by varying the type and amount of comonomer. LLDPEs are copolymers of ethylene and another 1-olefin, such as 1-butylene, 1-hexene, 4-methyl-1-pentene or 1-octene. Due to the intramolecular variation of comonomer content, the complex melting behaviour of LLDPE is of great interest (Schouterden *et al.*, 1985). LLDPE has superiority in properties compared to conventional LDPE, for example greater tensile strength and higher environmental stress crack resistance (Maddams and Woolmingon, 1985).

The last group of polyethylene is the very low density polyethylene (VLDPE) which comprises of only ethylene with α -olefins, such as 1-butene, 1-hexene or 1-octene and is recognized as having a high degree of linearity of structure with short chain branching rather than the long side branches characteristic of LDPE. Densities of VLDPEs are in the range of 0.900–0.915 g/cm³.

The density of the ethylene copolymers can be reduced into the elastomer range (0.865 – 0.90 g/cm³) by introducing even larger concentrations of comonomer. As the comonomer content of the polymer increases, and the density approaches 0.865 g/cm³, the tensile deformation changes from necking and cold drawing typical of a semi-crystalline thermoplastic to uniform drawing and high recovery characteristic of an elastomer. Copolymers with densities less than 0.9 g/cm³ have no lamellae or spherulites (Minick *et al.*, 1995).

2.1.2 Crosslinking of Polyethylene

A thermosetting PE has been obtained by a controlled process of crosslinking (vulcanization) (Al-Sammerai and Al-Nidawy, 1989). The material has enhanced stability at 105 °C or short term exposures at 230 °C and has a better resistance to environmental stress cracking. Crystallization is reduced by crosslinking and consequently modulus and hardness are lower. Polyethylenes may be crosslinked by the three different processes as follows:

1. Peroxide crosslinking is done by the incorporation of a peroxide with higher decomposition temperature such as dicumyl peroxide or a di – *t* -butyl peroxide.
2. Radiation crosslinking is done commercially on thin sections such as films.
3. Grafting of trialkoxyvinylsilane compounds onto the PE chain.

This study is based on the effects of radiation crosslinking on polymer blends. The effect of radiation on polymeric materials is an area of rapidly increasing interest. Several high technology industries require specialty polymers that exhibit a specific response upon exposure to radiation (Clough and Shalaby, 1991).

There are two types of radiation; high-energy and low-energy radiation. High-energy radiation includes gamma radiation, which is used for fundamental studies and for low-dose rate irradiation with deep penetration. The main sources for gamma radiation are the radioactive isotopes of cobalt-60 and cesium-137. For low-energy radiation, electron bombardment of suitable metal targets with electron

beams, or in a synchrotron, is used to produce X-radiation. Electron irradiation is normally obtained from electron accelerators to give beams with energies in the MeV range. The corresponding penetration depths are in the mm range.

The structural changes induced by irradiation can be traced by the chemical reactions involved. Most chemical transformations in polymers are originated from the formation of radicals. These appear due to the absorption of irradiation, which transfers energy to the polymer resulting in ionization, bond cleavages, and/or recombination reactions (Wintle, 1991). Even though the energy available largely exceeds the amount required to cleave any bonds, the bonds are not broken at random. Experimental evidence shows that selectivity rules, which do not follow bond energy considerations, may be applied (Chapiro, 1988). Therefore, in order to understand the effect of irradiation in the polymer, its final structure must be carefully studied.

Absorption of high-energy radiation by polymers produces excitation and ionization and it is these excited and ionized species that are the initial chemical reactants (Dole, 1973). The ejected electron must lose energy until it reaches thermal energy. The resultant excited molecule may undergo homolytic or heterolytic bond scission (Reichmanis *et al.*, 1993). Alternatively, the parent cation radical may undergo spontaneous decomposition, or ion-molecule reactions. The initially ejected electron may be stabilized by interaction with polar groups, as a solvated species or as an anion radical. Some chemical bonds and groups are particularly sensitive to radiation induced reactions. They include -COOH , C-X where X is a halogen, $\text{-SO}_2\text{-}$, NH_2 , and C=C .

The application of irradiation upon insulating polymeric materials may induce electrical conductivity and changes within the polymer structure. Such conductivity is related to the structural changes suffered by the polymer due mainly to the formation of radicals ions (Chapiro, 1988).

The molecular changes occurring in polymers as a result of radiation induced chemical reactions may be classified as:

1. Chain crosslinking effecting an increase in molecular weight and formation of a macroscopic network; polymer solubility decreases with radiation dose.
2. Chain scission effecting a decrease in molecular weight and, thus, substantially changing a polymer materials properties; strength, both tensile and flexural, decreases, and the rate of dissolution in a given solvent increases.

In addition to these changes, irradiation of polymers will frequently give rise to small molecule products, resulting from bond scission followed by abstraction or combination reaction.

2.2 Impact Modification with Polypropylene

Impact modification and rubber toughening of polypropylene has been researched intensively. So far, focus has been on the incorporation of ethylene-propylene-diene terpolymer (EPD), ethylene-propylene rubber (EPR) and styrene-ethylene/butylenes-

styrene triblock copolymers (SEBS) in polypropylene. Besides these, ethylene-vinyl acetate copolymers, polybutadiene and natural rubber have been studied as impact modifiers.

The subject of toughening mechanism in the area of rubber-toughened polymers has attracted interest. Based on the toughening mechanism in rubber toughened polymers, conditions for optimum impact strength are as follows (Bucknall, 1977):

1. The elastomer particles are finely and uniformly distributed in the plastics matrix.
2. The modulus of the elastomer is much less than that of the plastics.
3. The crystallinity of the elastomer is low.
4. A certain degree of interfacial adhesion is attained between the elastomer particles and the plastics matrix.
5. The cohesive strength of the elastomer is large.
6. A certain degree of entanglement of high-molecular weight polymer chain is attained in the plastics matrix.

2.3 Trimethylolpropane Triacrylate

Trimethylolpropane Triacrylate (TMPTA), $((\text{CH}_2\text{CHCOOCH}_2)_3\text{CC}_2\text{H}_5)$ trifunctional monomer, or also known as 1,1,1-Trimethylolpropane Triacrylate, 2-ethyl-2-[[[(1-oxoallyl)oxy]methyl]-1,3-propanediyl Diacrylate or 2-Propenoic acid 2-ethyl-2-[[[(1-oxo-2-propenyl)oxy]methyl]-1,3-propanediyl ester (Figure 2.1), is useful in the low volatility and fast cure response. It favors the property of resistance against weather, chemical, water and abrasion. It is used in manufacturing coatings, inks and